Calibration

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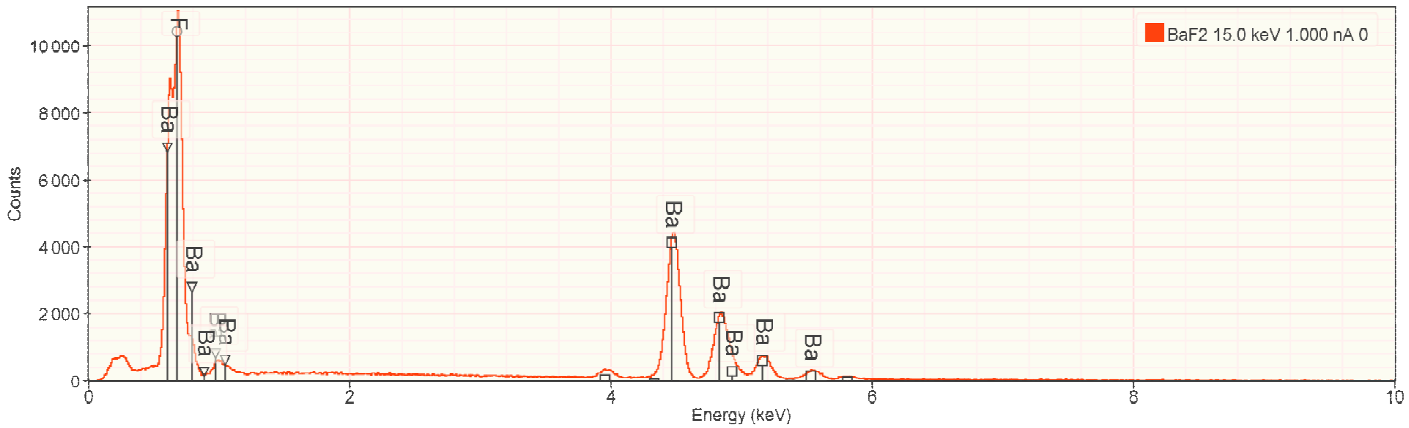
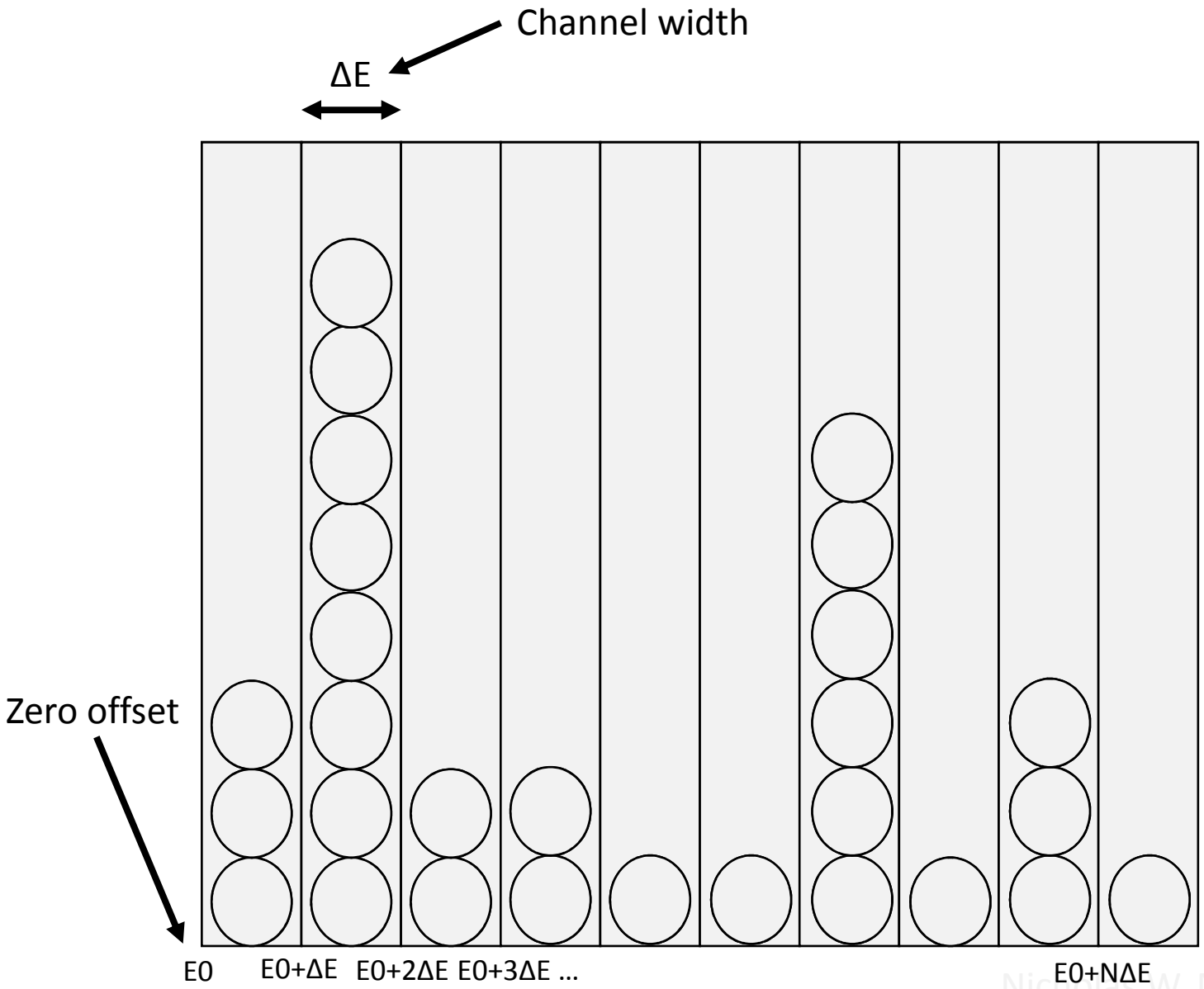


An x-ray spectrum is actually an accumulation of thousands to millions of individual x-ray event presented as a histogram. The x-axis of the histogram is a series of bins in which a number of x-ray events are accumulated. The width of each bin is typically nominally either 5 eV or 10 eV.

X-rays go into bins dependent on their energy…



Calibrated parameters

- The width of each bin ("channel width")
  - The width is typically expressed in eV/bin
- The offset of the 0^{th} bin ("zero offset")
  - The offset is typically expressed in eV.
  - Many detectors have a negative offset of a few hundred eV. Zero offset is also common.
- The resolution of the detector at Mn Kα ("FWHM").
  - The resolution is generally reported as the full-width-at-half-maximum (background corrected) for the Mn Kα peak.

These parameters can be extracted from a suitable measured spectrum

Calibrating what?

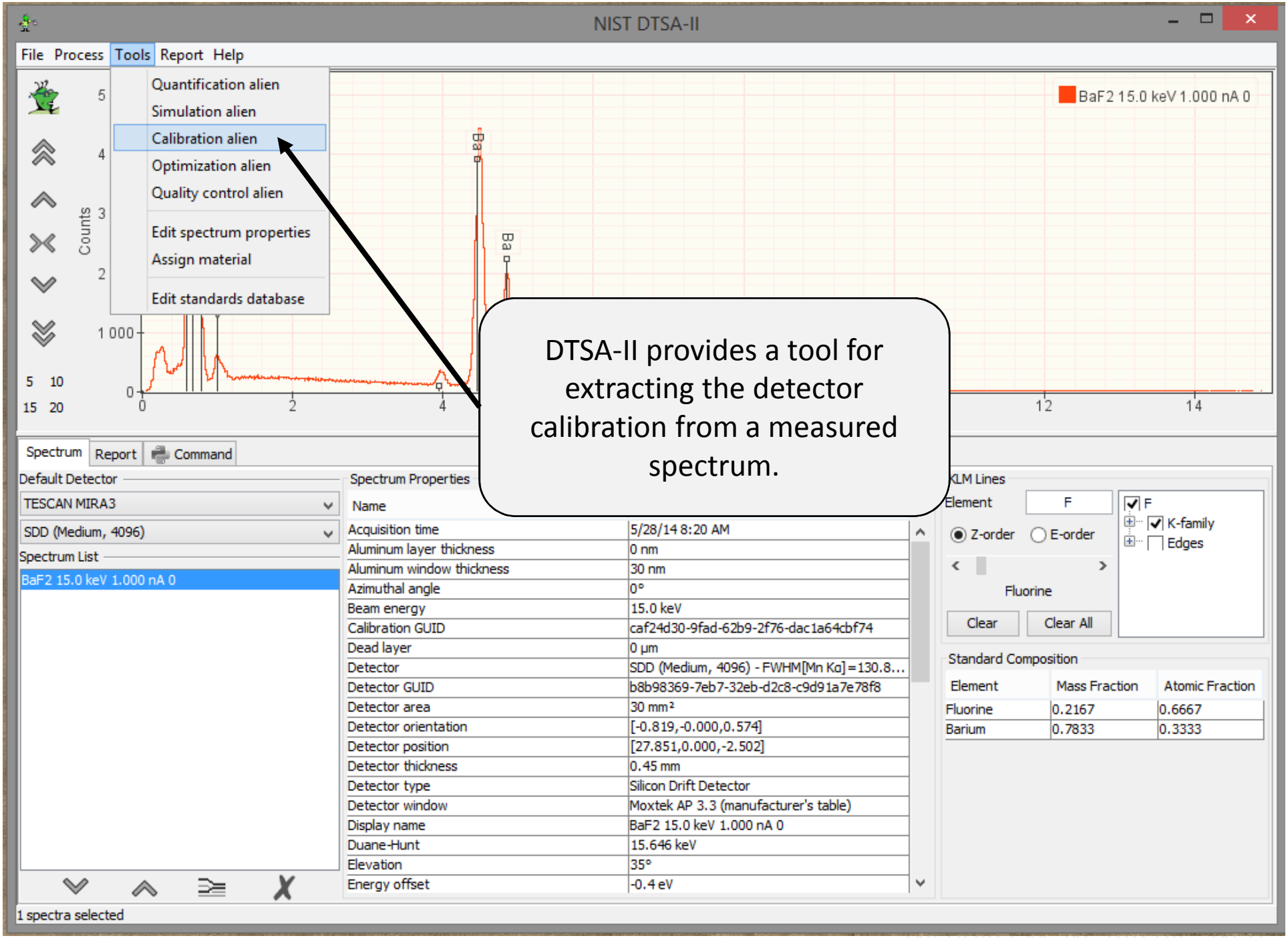
Although DTSA-II's detector model can compensate for a poorly calibrated detector, in practice, the detector should always be calibrated using the vendor's software.

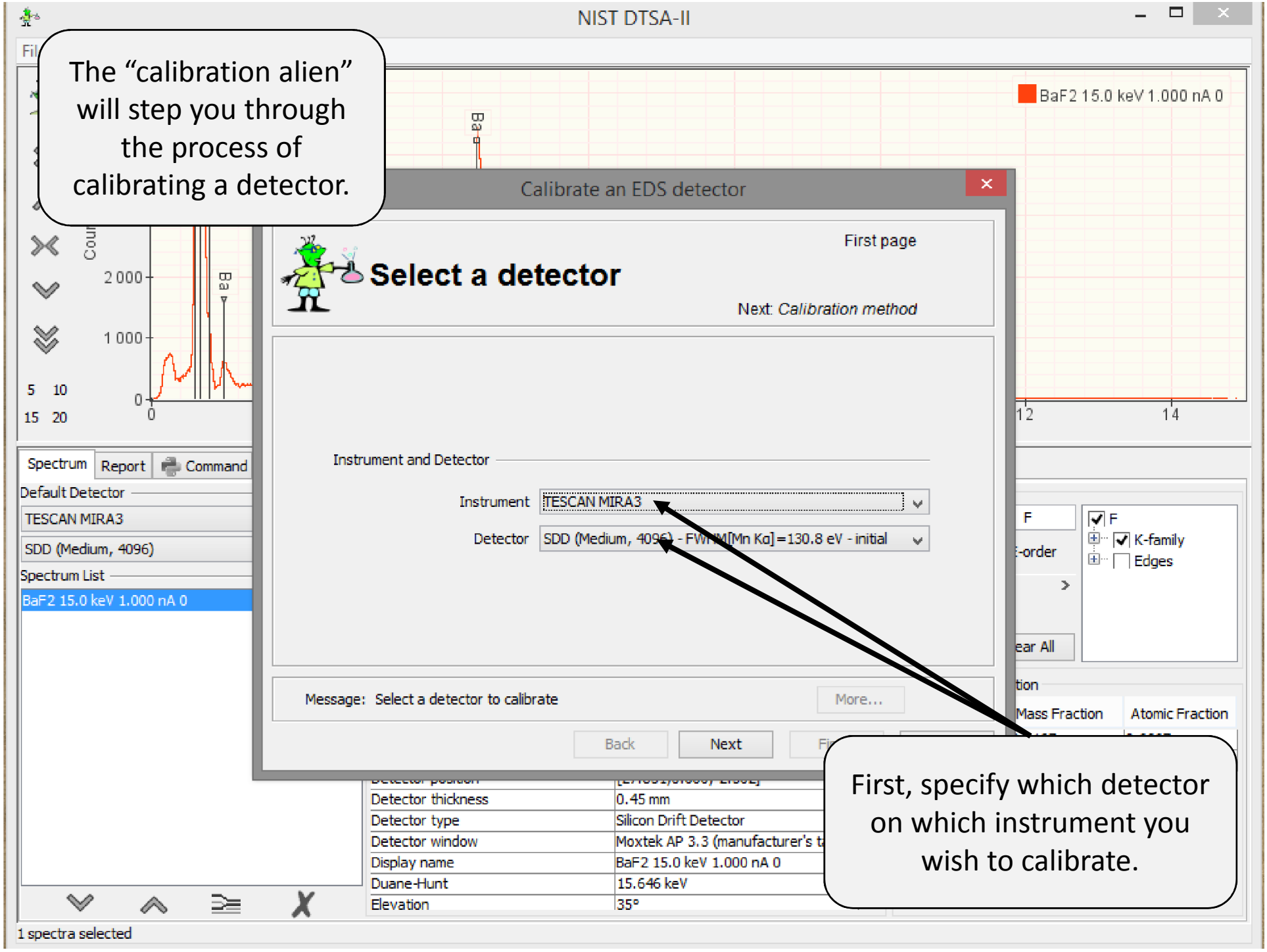
Calibrating the detector

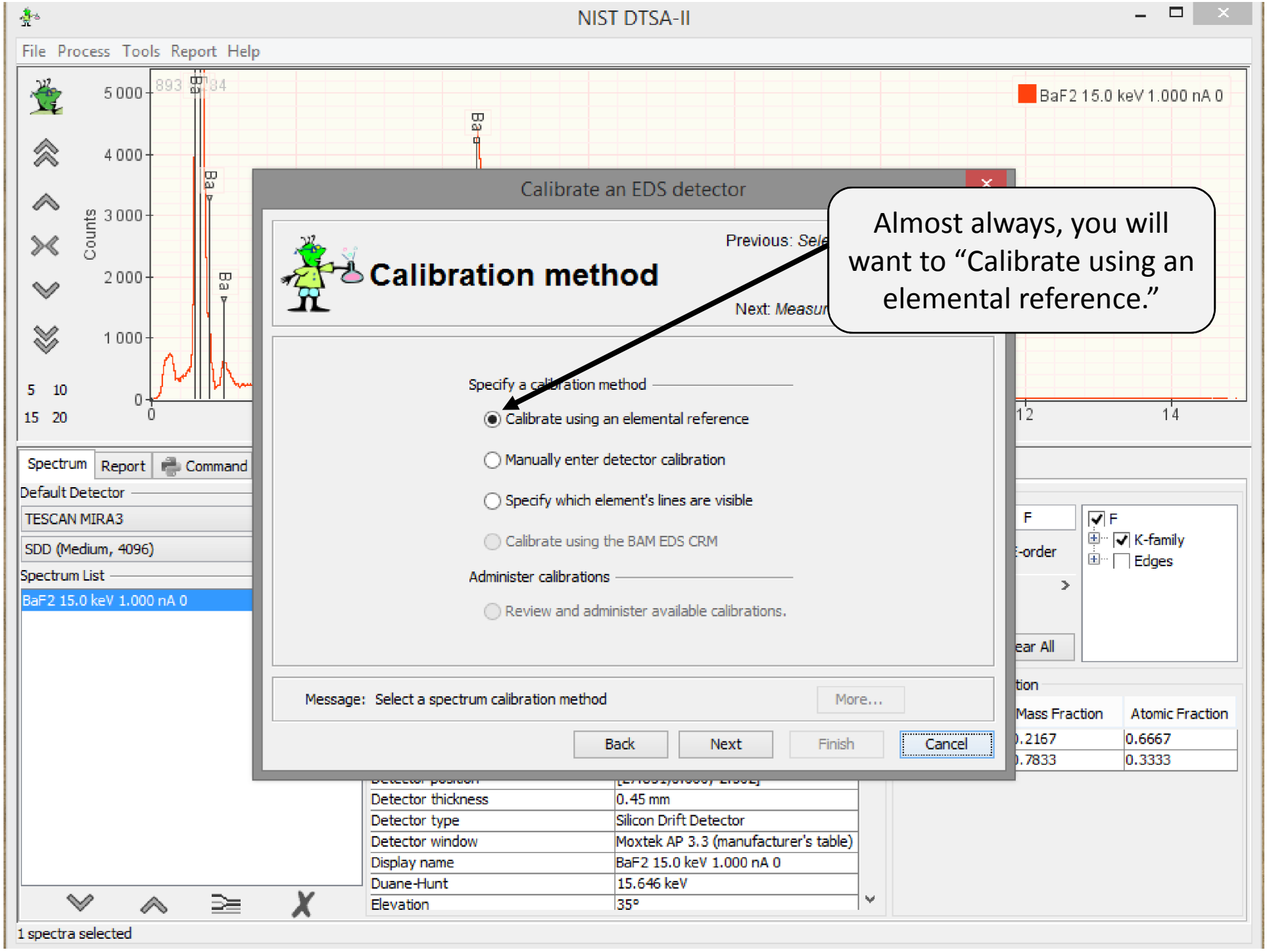
EDS detector vendors usually provide a mechanism for adjusting the hardware to produce a bin width close to the nominal value. On modern detectors the calibration is often performed automatically in the vendor software.

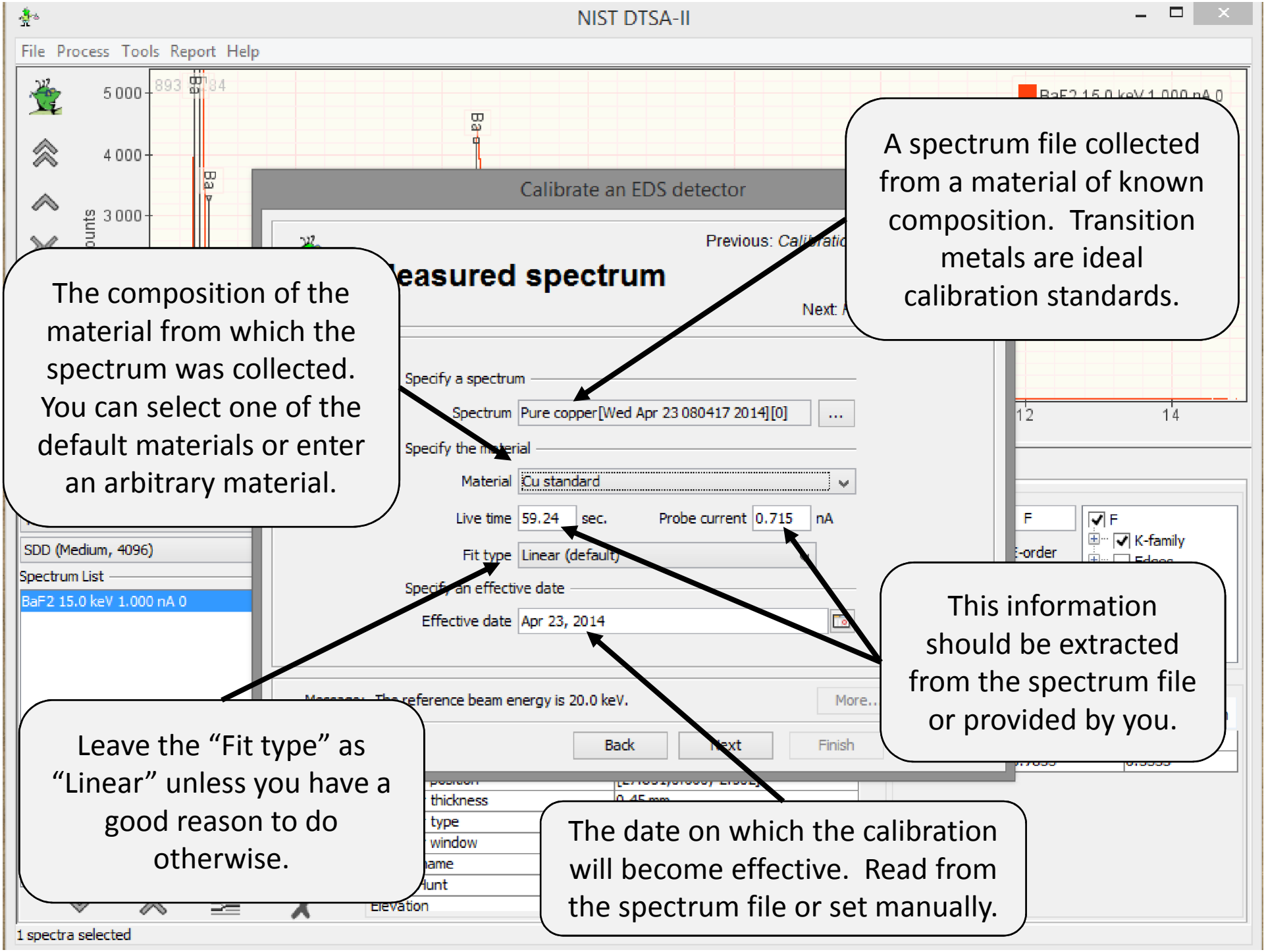
Calibrating the DTSA-II's detector model

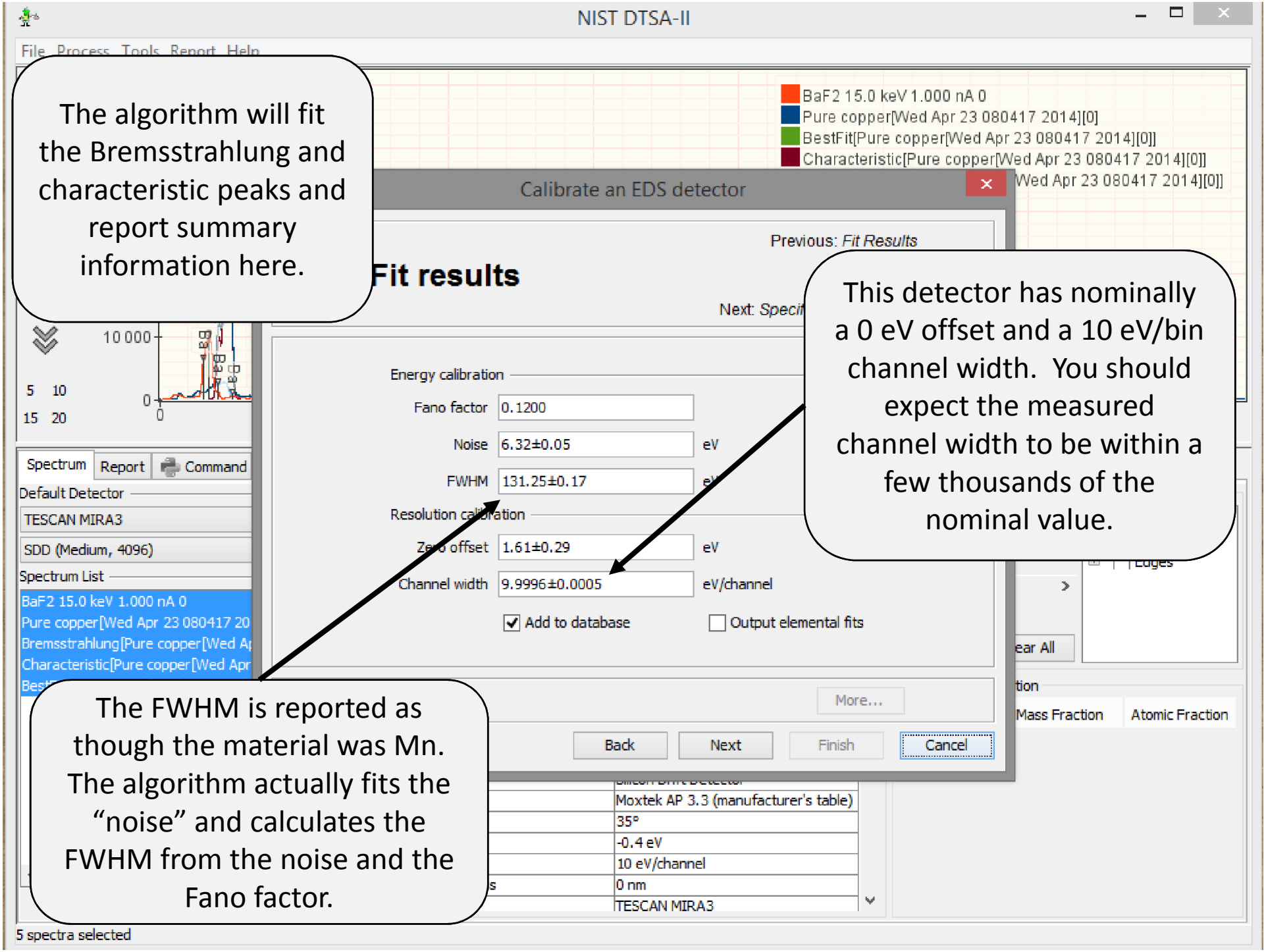
To interpret and simulate spectra, DTSA-II needs to know the real performance specifications of the detector. The calibration tool extracts this information from a measured spectrum.


NIST DTSA-II
File Process Tools Report Help
Quantification alien
Simulation alien
Calibration alien
Optimization alien
Quality control alien
Edit spectrum properties
Assign material
Edit standards database
BaF2 15.0 keV 1.000 nA 0
Counts
Ba
Ba
DTSA-II provides a tool for extracting the detector calibration from a measured spectrum.
Spectrum
Report
Command
Default Detector
TESCAN MIRA3
SDD (Medium, 4096)
Spectrum List
BaF2 15.0 keV 1.000 nA 0
Spectrum Properties
Name
Acquisition time 5/28/14 8:20 AM
Aluminum layer thickness 0 nm
Aluminum window thickness 30 nm
Azimuthal angle 0°
Beam energy 15.0 keV
Calibration GUID caf24d30-9fad-62b9-2f76-dac1a64cbf74
Dead layer 0 µm
Detector SDD (Medium, 4096) - FWHM[Mn Kα]=130.8...
Detector GUID b8b98369-7eb7-32eb-d2c8-c9d91a7e78f8
Detector area 30 mm²
Detector orientation [-0.819,-0.000,0.574]
Detector position [27.851,0.000,-2.502]
Detector thickness 0.45 mm
Detector type Silicon Drift Detector
Detector window Moxtek AP 3.3 (manufacturer's table)
Display name BaF2 15.0 keV 1.000 nA 0
Duane-Hunt 15.646 keV
Elevation 35°
Energy offset -0.4 eV
KLM Lines
Element F
Z-order
E-order
Fluorine
Clear
Clear All
F
K-family
Edges
Standard Composition
Element Mass Fraction Atomic Fraction
Fluorine 0.2167 0.6667
Barium 0.7833 0.3333
1 spectra selected


NIST DTSA-II
The “calibration alien” will step you through the process of calibrating a detector.
BaF2 15.0 keV 1.000 nA 0
Calibrate an EDS detector
First page
Select a detector
Next: Calibration method
Instrument and Detector
Instrument
TESCAN MIRA3
Detector
SDD (Medium, 4096) - FWHM[Mn Kα]=130.8 eV - initial
Message: Select a detector to calibrate
More...
Back
Next
First, specify which detector on which instrument you wish to calibrate.
Spectrum
Report
Command
Default Detector
TESCAN MIRA3
SDD (Medium, 4096)
Spectrum List
BaF2 15.0 keV 1.000 nA 0
Detector thickness
0.45 mm
Detector type
Silicon Drift Detector
Detector window
Display name
BaF2 15.0 keV 1.000 nA 0
Duane-Hunt
15.646 keV
Elevation
35°
Mass Fraction
Atomic Fraction
K-family
Edges
1 spectra selected


NIST DTSA-II
File Process Tools Report Help
BaF2 15.0 keV 1.000 nA 0
Counts
Calibrate an EDS detector
Calibration method
Previous: Sele
Next: Measur
Specify a calibration method
Calibrate using an elemental reference
Manually enter detector calibration
Specify which element's lines are visible
Calibrate using the BAM EDS CRM
Administer calibrations
Review and administer available calibrations.
Message: Select a spectrum calibration method
More...
Back
Next
Finish
Cancel
Almost always, you will want to "Calibrate using an elemental reference."
Spectrum
Report
Command
Default Detector
TESCAN MIRA3
SDD (Medium, 4096)
Spectrum List
BaF2 15.0 keV 1.000 nA 0
F
K-family
Edges
Mass Fraction
Atomic Fraction
0.2167
0.6667
0.7833
0.3333
Detector thickness
0.45 mm
Detector type
Silicon Drift Detector
Detector window
Moxtek AP 3.3 (manufacturer's table)
Display name
BaF2 15.0 keV 1.000 nA 0
Duane-Hunt
15.646 keV
Elevation
35°
1 spectra selected

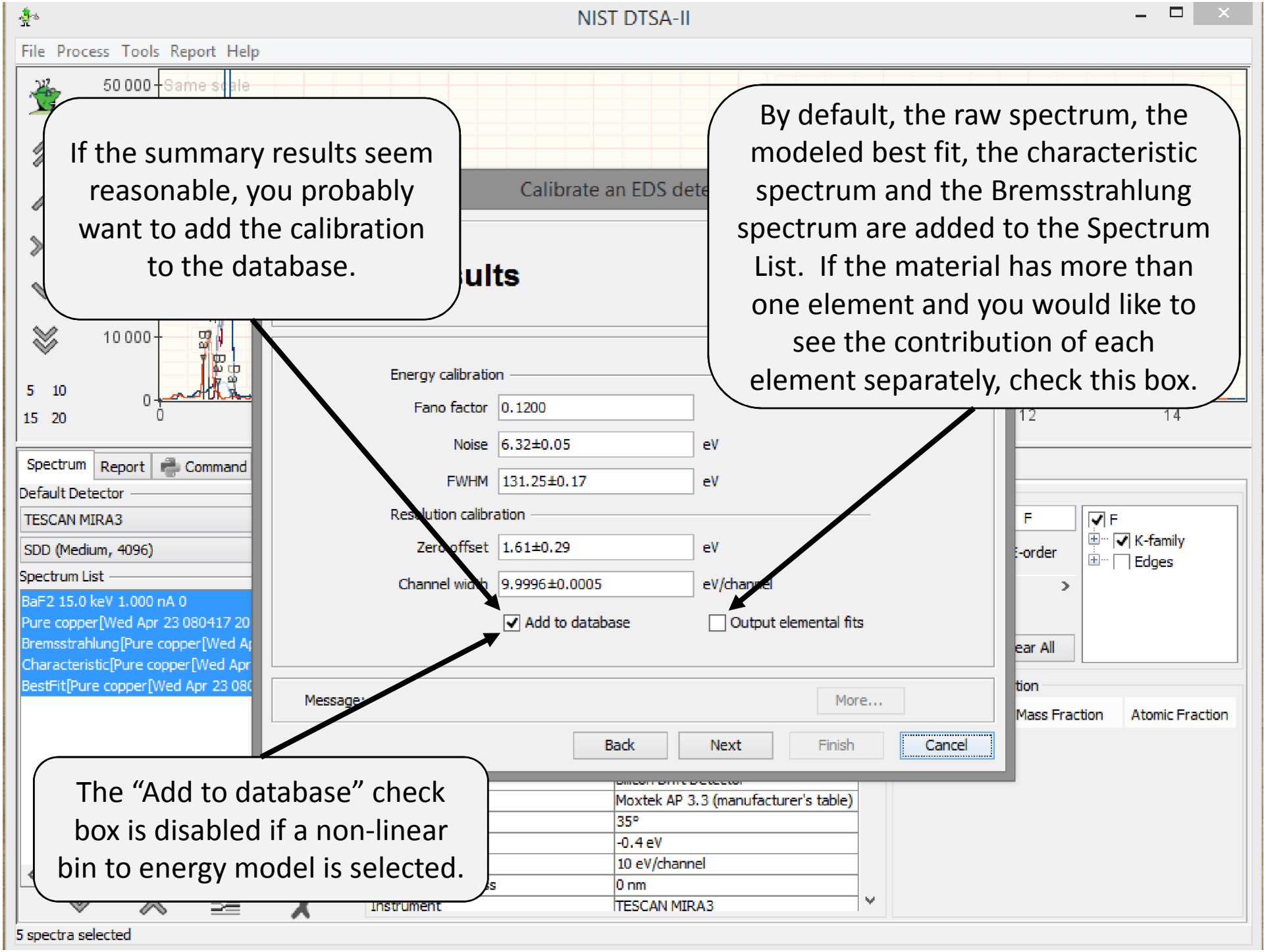

NIST DTSA-II
File Process Tools Report Help
Calibrate an EDS detector
Specify a spectrum
Spectrum Pure copper[Wed Apr 23 080417 2014][0]
Specify the material
Material Cu standard
Live time 59.24 sec.
Probe current 0.715 nA
Fit type Linear (default)
Specify an effective date
Effective date Apr 23, 2014
The reference beam energy is 20.0 keV.
More..
Back
Next
Finish
SDD (Medium, 4096)
Spectrum List
BaF2 15.0 keV 1.000 nA 0
1 spectra selected
A spectrum file collected from a material of known composition. Transition metals are ideal calibration standards.
The composition of the material from which the spectrum was collected. You can select one of the default materials or enter an arbitrary material.
This information should be extracted from the spectrum file or provided by you.
Leave the “Fit type” as “Linear” unless you have a good reason to do otherwise.
The date on which the calibration will become effective. Read from the spectrum file or set manually.

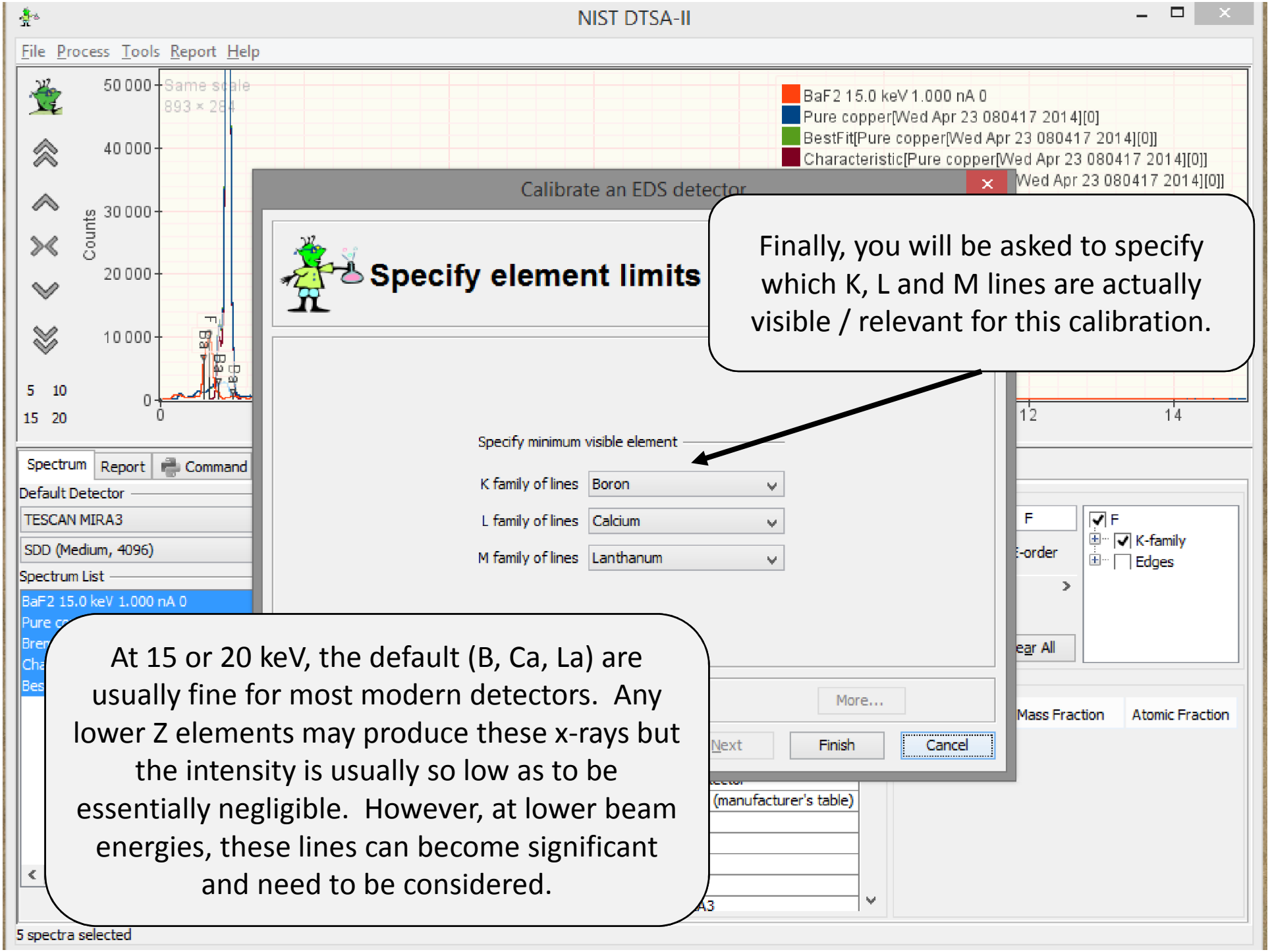


The algorithm will fit the Bremsstrahlung and characteristic peaks and report summary information here.

This detector has nominally a 0 eV offset and a 10 eV/bin channel width. You should expect the measured channel width to be within a few thousands of the nominal value.

The FWHM is reported as though the material was Mn. The algorithm actually fits the "noise" and calculates the FWHM from the noise and the Fano factor.

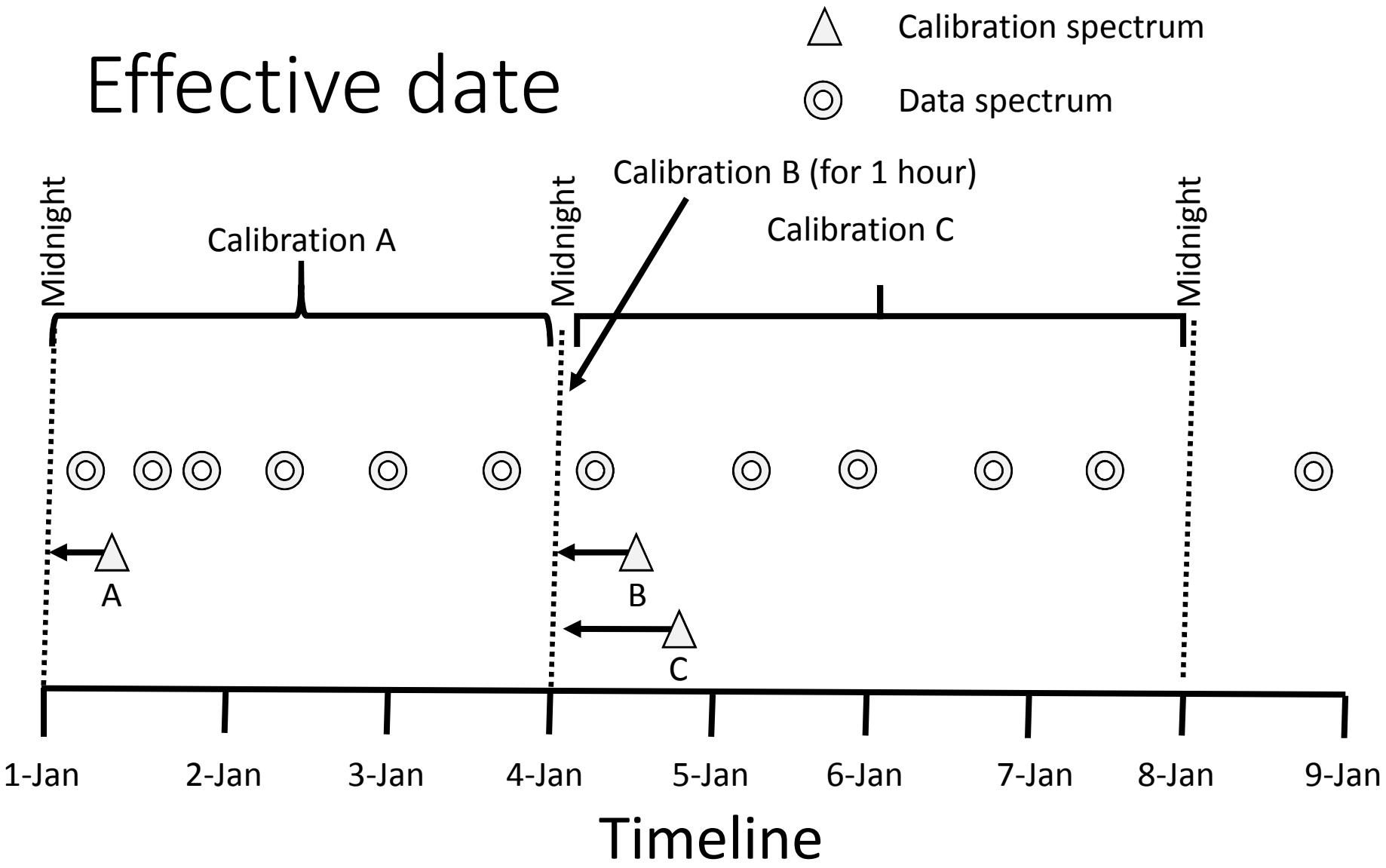

NIST DTSA-II
File Process Tools Report Help
If the summary results seem reasonable, you probably want to add the calibration to the database.
By default, the raw spectrum, the modeled best fit, the characteristic spectrum and the Bremsstrahlung spectrum are added to the Spectrum List. If the material has more than one element and you would like to see the contribution of each element separately, check this box.
Energy calibration
Fano factor 0.1200
Noise 6.32±0.05 eV
FWHM 131.25±0.17 eV
Resolution calibration
Zero offset 1.61±0.29 eV
Channel width 9.9996±0.0005 eV/channel
Add to database
Output elemental fits
Message:
More...
Back
Next
Finish
Cancel
Spectrum
Report
Command
Default Detector
TESCAN MIRA3
SDD (Medium, 4096)
Spectrum List
BaF2 15.0 keV 1.000 nA 0
Pure copper[Wed Apr 23 080417 20
Bremsstrahlung[Pure copper[Wed Ap
Characteristic[Pure copper[Wed Apr
BestFit[Pure copper[Wed Apr 23 080
Mass Fraction
Atomic Fraction
Moxtek AP 3.3 (manufacturer's table)
35°
-0.4 eV
10 eV/channel
0 nm
TESCAN MIRA3
The “Add to database” check box is disabled if a non-linear bin to energy model is selected.
5 spectra selected


NIST DTSA-II
File Process Tools Report Help
Same scale
893 × 284
BaF2 15.0 keV 1.000 nA 0
Pure copper[Wed Apr 23 080417 2014][0]
BestFit[Pure copper[Wed Apr 23 080417 2014][0]]
Characteristic[Pure copper[Wed Apr 23 080417 2014][0]]
Counts
Calibrate an EDS detector
Specify element limits
Specify minimum visible element
K family of lines Boron
L family of lines Calcium
M family of lines Lanthanum
More...
Next
Finish
Cancel
Finally, you will be asked to specify which K, L and M lines are actually visible / relevant for this calibration.
At 15 or 20 keV, the default (B, Ca, La) are usually fine for most modern detectors. Any lower Z elements may produce these x-rays but the intensity is usually so low as to be essentially negligible. However, at lower beam energies, these lines can become significant and need to be considered.
Spectrum
Report
Command
Default Detector
TESCAN MIRA3
SDD (Medium, 4096)
Spectrum List
BaF2 15.0 keV 1.000 nA 0
Mass Fraction
Atomic Fraction
K-family
Edges
5 spectra selected

What is the “Effective date?”

- Each data spectrum is associated with the most recent prior calibration as it is read in.
- The assumption is that the calibration spectrum is collected before or at least on the same day as the data spectra. You can fake this by manually changing the effective date.
- A data spectrum will be associated with the most recent calibration.
- Calibrations are pre-dated to take effect at 12:01 AM on their “effective date.” Each subsequent calibration collected on a day is assumed to take effect 1 hour later than the previous one.

Effective date



Timeline

The first calibration on a day becomes effective at 12:01 AM
The second becomes effective at 1:01 AM, third at 2:01 AM etc.

How is the calibration used?

- Quantification
  - To determine the position and width of the region to fit for each characteristic x-ray transition.
- Simulation
  - To determine the bins into which the simulated Bremsstahlung and characteristic x-rays will be placed.
  - To determine the simulated width of the characteristic x-ray peaks.
- KLM lines
  - Ensures the peaks line up with the KLM lines

Advanced technique

The “calibration alien” “calibrate using standard spectrum” tool can actually fit spectra from arbitrary materials.

Use 1: This can be useful when a standard requires a reference but a good reference can not be found. You can use the “output elemental fits” to export “characteristic-only spectra” for any element which can be used as either a reference or a standard.

Use 2: The elemental fits can also be used to subtract off undesired characteristic peaks.